

NOV 02 2006

Patent  
Docket No.: 54538US011

## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

First Named Inventor:	KENNETH L. SMITH	Group Art Unit:	1733
Application No.	09/870,180	Confirmation No.	7800
Filed:	05/30/2001	Examiner:	John L. Goff II

Title: CUBE CORNER CAVITY BASED RETROREFLECTORS WITH  
TRANSPARENT FILL MATERIAL.

DECLARATION UNDER 37 C.F.R. 1.132 OF BIMAL V. THAKKAR

I, Bimal V. Thakkar, hereby declare that:

1. I received a B.S. in Chemistry from the University of Bombay, India in 1982, a B.S. in Chemical Engineering from Northwestern University in Evanston, Illinois in 1985, and a Ph.D. in Chemical Engineering from the University of Minnesota in Minneapolis, Minnesota in 1991. My graduate studies focused on polymer surface science.
2. I am an employee of 3M Company ("3M"). 3M IPC, the assignee of the above-identified application, is a subsidiary of 3M. 3M is also the exclusive licensee of all patents owned by 3M IPC. I have been an employee of 3M since June of 1991. From June of 1991 through the present, I have been working in Traffic Safety Systems Division, and I have continuously worked in the area of pressure sensitive adhesive formulations and silicone release liners. My current title is Senior Research Specialist.
3. I identified Ebecryl® 3720 Bisphenol A Epoxy Diacrylate, commercially available from Cytec Surface Specialties, Inc., Smyrna, GA. (hereinafter "Ebecryl 3720"), as a material that has both acrylate and epoxy functionality and, to my knowledge, that does not include other monomers. The product literature for Ebecryl 3720 is attached to this Declaration as Exhibit I.

4. Pages 216-217 of *Adhesion and Adhesives Technology, An Introduction* states that "The Pressure-Sensitive Tape Council has defined pressure-sensitive adhesives as materials with the following properties:

1. Aggressive and permanent tack
2. Adheres with no more than finger pressure
3. Requires no activation by any energy source
4. Has sufficient ability to hold onto the adherend
5. Has enough cohesive strength to be able to be removed cleanly from the adherend."

(Alphonsus V. Pocius, Hanser Publishers, New York, 1997 at Chapter 9, pp. 216-217, Paragraph 9.2, quoting *Test Methods for Pressure Sensitive Adhesive Tapes* (1994), Pressure Sensitive Tape Council, Chicago, IL). A copy of the quoted portion of *Adhesion and Adhesives Technology, An Introduction* is attached to this Declaration as Exhibit II.

5. The Pressure-Sensitive Tape Council has at least one test that is useful in determining whether and to what extent a material is a pressure-sensitive adhesive. This test is referred to as the PSTC Test (Pressure-Sensitive Tape Council Test).

6. The PSTC Test was replaced by the ASTM Test Method D 3654 test (see the attached Exhibit III at paragraph 1.2.2, which is a copy of the ASTM Test D 3654).

7. In order to determine whether uncured Ebercyl 3720 satisfies the requirement that the material have "sufficient ability to hold onto the adherend," I employed ASTM Test Method D 3654 entitled "Standard Test Methods for Shear Adhesion of Pressure-Sensitive Tapes" (see Exhibit III).

8. The Shear Adhesion to Standard Steel Panel Test (Procedure A of ASTM Test Method D 3654) involves applying "[a] strip of tape . . . to a standard steel panel under controlled roll down. The panel is mounted vertically, a standard mass is attached to the free end of the tape and the time to failure is determined." (See page 2, Section 3.1 of Exhibit III).

9. I prepared two samples by cutting two 12.7 mm wide by 10 cm long strips of 3M™ Diamond Grade™ VIP Reflective Sheeting Series 3990. I applied

2-3 drops of Ebecryl 3720 to the non-retroreflective side of a 12.7 mm by 12.7 mm area at one end of each sample. Each sample was placed on a new 2 1/4 inch square stainless steel panel such that the 12.7 mm by 12.7 mm area onto which the Ebecryl 3720 had been applied was in contact with the stainless steel panel and was aligned in the center and at the bottom edge of the stainless steel panel. Contact between the Ebecryl 3720 and the panel was established using one back and forth motion with a 2 pound rubber roller. The portion of each sample extending below the stainless steel panel was folded back on itself to form a loop and secured with a staple as is shown and described in Figure 1 of ASTM Test Method D 3654.

10. The first sample was placed vertically on a test fixture as is shown and described in Figure 1 of ASTM Test Method D 3654 (Exhibit III). Within 30 minutes, this sample slid off the stainless steel panel before the weight was attached to the sample.

11. The second sample was placed horizontally on a countertop and was conditioned for 24 hours at room temperature and about 50% relative humidity. After 24 hours of conditioning, the second sample was placed vertically on a test fixture as shown and described in Figure 1 of ASTM Test Method D 3654 (Exhibit III). A 1000 gram weight was attached to the looped end of the sample. The sample fell off the panel immediately.

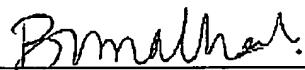
12. In summary, the results of the above tests were that the first sample slid off the panel before the weight was applied and the second sample slid off the panel immediately upon application of the weight. Commonly recognized pressure-sensitive adhesives tested under the same conditions as detailed in above are capable of holding the weight for hundreds to thousands of minutes before the sample separates from the steel panel. Consequently, Ebecryl 3720 does not satisfy the Pressure-Sensitive Tape Council's requirement that the material have "sufficient ability to hold onto the adherend" and thus uncured Ebecryl 3720 does not qualify as a pressure-sensitive adhesive.

13. The Ebecryl 3720 product literature states that the UV or EB cured Ebecryl 3720 product has "[h]igh surface hardness" and lists under "Typical Cured Properties" a Tensile strength of 11,000 psi. 11,000 psi converts to  $7.6 \times 10^8$  dynes/cm<sup>2</sup>. The Dahlquist criteria described in the *Handbook of Pressure Sensitive*

*Adhesives Technology*, D. Satas, 3rd ed. Page 191 (1999) (attached as Exhibit IV) states that "Dahlquist's criterion suggests that the compressive modulus of a pressure sensitive adhesive cannot exceed  $10^7$  dynes/cm<sup>2</sup> in the time frame of the bonding process." Consequently, Ebecryl 3720 in the cured state does not qualify as a pressure sensitive adhesive.

14. In summary, Ebecryl 3720 is an acrylic based epoxy material not including other monomers, and Ebecryl 3720 does not qualify as a pressure-sensitive adhesive in either its uncured or cured form.

15. I declare that all statements made herein are of my own knowledge, are true, and that all statements made on information and belief are believed to be true. I further declare that these statements were made with the knowledge that willful, false statements and the like so made are punishable by fine or imprisonment or both, under 18 USC 1001, and that such willful, false statements may jeopardize the validity of the patent application or any patent issuing thereon.



Bimal V. Thakkar

Subscribed and sworn to before me  
this 2nd day of Nov, 2006.

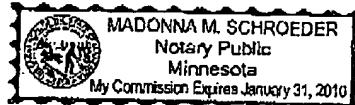
  
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Notary Public

EXHIBIT 1



## EBECRYL® 3720

Bisphenol A Epoxy Diacrylate

### INTRODUCTION

EBECRYL® 3720 is a bisphenol A epoxy diacrylate that is the industry standard in performance and consistency. It exhibits light color and fast cure response. Films of EBECRYL 3720 cured via exposure to ultraviolet light (UV) or electron beam (EB) demonstrate high surface hardness, gloss and excellent chemical resistance. EBECRYL 3720 finds broad use in UV/EB applications, such as inks, coatings, and overprint varnishes and offers improved formulation stability.

### PERFORMANCE HIGHLIGHTS

EBECRYL 3720 is characterized by:

- Light color
- Fast cure response
- Good stability

UV/EB cured properties based on EBECRYL 3720 are characterized by the following performance properties:

- High gloss
- Excellent chemical resistance
- High surface hardness

The actual properties of UV/EB cured products also depend on the selection of other formulation components such as reactive diluents, additives and photoinitiators.

### SUGGESTED APPLICATIONS

Formulated UV/EB curable products containing EBECRYL 3720 may be applied via direct or reverse roll, offset gravure, metering rod, slot die, knife over roll, air knife, curtain, immersion and spin coating methods, as well as screen printing. EBECRYL 3720 is recommended for use in:

- Paper upgrading
- Overprint varnishes
- Screen inks
- Wood sealers
- Fast cure coatings
- Laminating adhesives
- Coatings for paper, paperboard, wood, chipboard and rigid plastics

### SPECIFICATIONS

	SMT <sup>(1)</sup>	Value
Color, Gardner scale, max.	001-B	2
Appearance	002-A	Clear liquid
Acid value, mg KOH/g, max.	009-L	2
Viscosity at 65.5°C, cP	013-CC	1500-2500
% Epoxydc, max.	065-M	0.5

### TYPICAL PHYSICAL PROPERTIES

Density, g/ml at 25°C	1.13
Functionality, theoretical <sup>(2)</sup>	2
Oligomer, % by weight	100

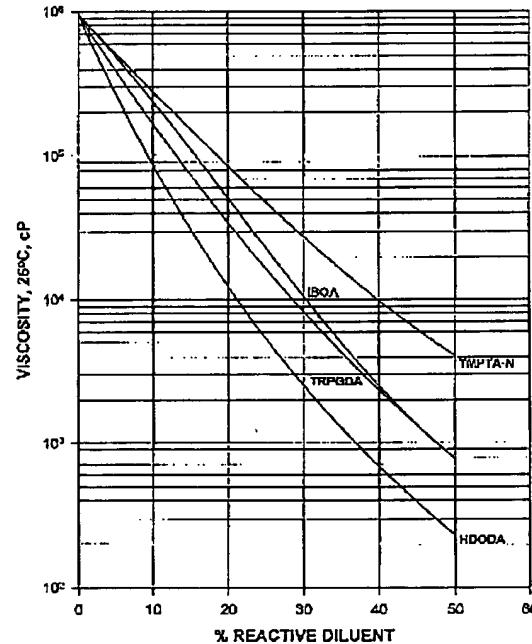
### TYPICAL CURED PROPERTIES<sup>(3)</sup>

Tensile strength, psi	11000
Elongation at break, %	8
Young's modulus, psi	204000
Glass transition temperature, °C <sup>(4)</sup>	67

### Graph I

### EBECRYL 3720

### Viscosity Reduction with Reactive Diluents



\* EBECRYL UV curable resins and oligomers is a registered trademark of Cytec Surface Specialties Inc.

(1) Standard Methods of Testing available upon request.

(2) Theoretical determination based on the undiluted oligomer.

(3) UV cured 125 µ thick films.

(4) Determined by Dynamic Mechanical Analysis.

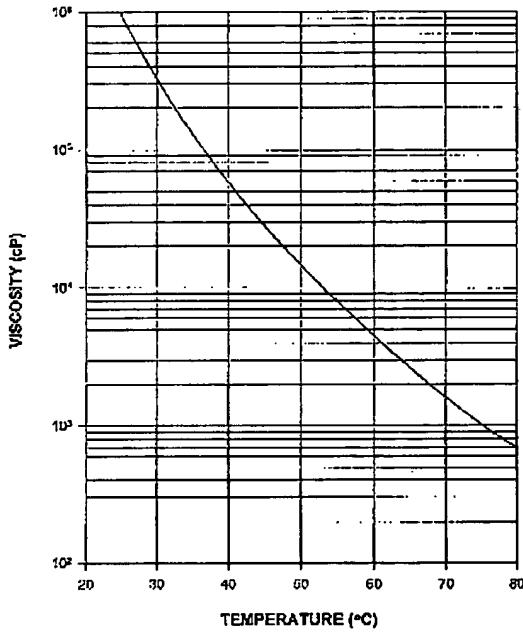


#### VISCOOSITY REDUCTION

Graph I shows the viscosity reduction of *EBECRYL* 3720 with 1,6-hexanediol diacrylate (HDDA)<sup>(1)</sup>, isobornyl acrylate (IBOA)<sup>(1)</sup>, trimethylolpropane triacrylate (TMPTA-N)<sup>(1)</sup>, and tripropylene glycol diacrylate (TRPGDA)<sup>(1)</sup>. Although viscosity reduction can be achieved with non-reactive solvents, reactive diluents are preferred because they are essentially 100 percent converted during UV/EB exposure to form a part of the coating or ink, thus reducing solvent emissions. The specific reactive diluents used will influence performance properties such as hardness and flexibility.

Graph II illustrates the change in viscosity of *EBECRYL* 3720 with increasing temperature.

**Graph II**  
***EBECRYL* 3720**  
Viscosity vs. Temperature



(1) Product of Cytec Surface Specialties.

#### STORAGE AND HANDLING

Before using *EBECRYL* 3720, consult the Material Safety Data Sheet for additional information on hazards, handling procedures, and recommended protective equipment.

The maximum recommended storage temperature for *EBECRYL* 3720 is 38°C (100°F). High temperature and fire conditions can cause uncontrolled polymerization with rapid evolution of heat and pressure rise, which may result in violent rupture of the storage tanks or containers. Never store in direct sunlight or adjacent to heated compartments. Containers should be kept closed and away from oxidizing agents, acids, alkalies, peroxides, free radical initiators, photosensitizers, rust, and x-ray or ultraviolet radiation. Procedures that displace oxygen from the material, such as sparging with nitrogen, should be avoided.

#### PRECAUTIONS

Avoid contact with skin and eyes and breathing vapors. Contains materials that may cause irritation to the eyes and skin. Sensitization may occur. Skin irritation may not occur immediately and contact may go unnoticed for up to 48 hours. Solvents should not be used to clean skin because of increased penetration potential. Contaminated clothing, shoes, belts and other leather goods should be removed immediately. Incinerate contaminated leather goods, including shoes. Wash contaminated clothing thoroughly before reuse.

Please refer to the Cytec Surface Specialties **Guide to Safety and Handling** for additional information on the safe handling of acrylates.

• Cytec Surface Specialties Inc. | 1950 Lake Park Drive, Smyrna GA 30080 | Phone 800-433-2873 | Fax 678-255-4699  
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*EBECRYL* 3720 03/2005

EXHIBIT II

Alphonsus V. Pocius

# Adhesion and Adhesives Technology

An Introduction



Hanser Publishers, Munich Vienna New York

Hanser/Gardner Publications, Inc., Cincinnati

EXHIBIT II

## The Author:

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EXHIBIT II

## 9 The Chemistry and Physical Properties of Elastomer-Based Adhesives

### 9.1 Introduction

Of the classes of adhesives, elastomer-based adhesives are probably the most familiar to the consumer. Many of the baby boom generation remember solvent-thinned, rubber-based paper adhesives used for elementary school projects. Many of the pieces of furniture we have in our homes and offices are laminated wood. The adhesive used for lamination is most often based on an elastomer. Probably the most widely recognized elastomer-based adhesive is coated on a backing and used as a pressure sensitive adhesive tape. In this chapter, the chemistry and physical properties of pressure-sensitive adhesives as well as other types of elastomer-based adhesives is discussed. The physical properties of pressure-sensitive adhesives is emphasized as these demonstrate many of the concepts discussed in this book. Other elastomer-based adhesives are discussed in terms of their chemistry, because fundamental information on these materials is limited.

The objectives of this chapter include the development of an understanding of the chemistry of elastomer-based adhesives. Knowledge of the parameters necessary to formulate this type of adhesive and an appreciation of the test methods evaluating pressure-sensitive adhesives should be gained. Most important is the discussion on how the dynamic mechanical properties of pressure-sensitive adhesives are related to their mechanism of action.

### 9.2 Pressure-Sensitive Adhesives

The Pressure-Sensitive Tape Council [1] has defined pressure-sensitive adhesives as materials with the following properties:

1. Aggressive and permanent tack
2. Adheres with no more than finger pressure
3. Requires no activation by any energy source
4. Has sufficient ability to hold onto the adherend
5. Has enough cohesive strength to be able to be removed cleanly from the adherend

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## 9.2 Pressure-Sensitive Adhesives 217

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The above definition of a pressure-sensitive adhesive (PSA) includes some concepts yet to be discussed but that are extremely important for this class of materials. The property we first notice about PSAs is their *tack*. We know what tack feels like but how do we describe it and, for that matter, how do we generate materials with tack? Tack is generated by adding certain low molecular weight materials to elastomers in a process called *tackification*. These *tackifiers*, because of their low molecular weight, decrease the cohesive strength of the elastomer. However, a PSA must have tack and sufficient cohesive strength to hold two things together. In this section, we see how a balance of properties is generated so that a PSA combines these seemingly mutually exclusive properties to yield a material with "sufficient ability to hold onto the adherend" as well as "be cleanly removed from the adherend."

For the most part, PSAs are used in coated form as the adhesives in pressure-sensitive adhesive tapes (PSAT's). There is substantial technology associated with the manufacture of pressure-sensitive tapes but much of the information regarding PSAT's is proprietary to those companies which manufacture them. The information about manufacturing does not yield more understanding as to how these materials perform and therefore, it is not discussed in this book.

### 9.2.1 Chemistry of the Base Resins Used in PSAs

A large variety of elastomers have been used as PSAs. The first material to gain widespread use was a natural rubber-based adhesive. Natural rubber is poly(*cis*-isoprene) and is obtained from the *Hevea* rubber plant as a natural latex. The structure of poly(*cis*-isoprene) is shown in Fig. 9.1. The latex is coagulated and then the rubber is usually smoked to eliminate bacteria and fungi which can degrade the rubber before it can be processed. The PSA manufacturer receives the natural rubber as a slab of smoked material which is often worked mechanically to reduce its molecular weight. The mechanically worked rubber is then dissolved in an appropriate solvent, mixed with the tackifier and coated or otherwise packaged. Natural rubber-based PSAs are still used in a number of PSAT applications including masking tape where they exhibit excellent removability after painting and baking. One of the primary attributes of natural rubber-based PSAs is their low cost, but they also are used extensively because of their high peel strength when properly formulated. The properties of natural rubber-based PSA lead to most of the discussion on PSA performance, which follows in a later section.

Natural rubber-based PSAs were the basis for all of the early PSA products. However, these adhesives had one primary flaw. Because of unsaturation in the backbone of the base polymer, the adhesive had a noticeable tendency to yellow and to crosslink, thus becoming brittle. The problem could in some ways be ameliorated by the addition of antioxidants, but, in general, natural rubber-based PSAs were unstable to long term exposure to the environment. This problem led to the introduction of a number of new base resins which did not suffer from these deficiencies.

EXHIBIT II

## References 245

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9. (a) Gerrard, J.A., and Mattson, R.C., U.S. Patent 2,918,112 (1959). (b) J. Kermadjian, *Adhesive Age*, 5(6) (1962), p. 34

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## EXHIBIT III



Designation: D 3654/D 3654M - 06

## Standard Test Methods for Shear Adhesion of Pressure-Sensitive Tapes<sup>1</sup>

This standard is issued under the fixed designation D 3654/D 3654M; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

*This standard has been approved for use by agencies of the Department of Defense.*

### 1. Scope

1.1 This test method covers procedures for determining the ability of pressure-sensitive tapes and labels to remain adhered under constant load applied parallel to the surface of the tape and substrate.

1.1.1 Procedure A measures the shear adhesion when applied to a vertical standard steel panel.

1.1.2 Procedure B measures the shear adhesion when applied to vertical panel covered with NIST SRM 1810A standard fiberboard.

1.1.3 Procedure C measures the shear adhesion when applied to a vertical panel covered with a fiberboard as defined by Comité Européen de Normalisation (CEN).

1.1.4 Procedure D measures shear adhesion when applied to a vertical panel covered with a fiberboard agreed upon by the buyer and seller.

1.1.5 Procedure E measures shear adhesion of filament reinforced tape when applied to a horizontal standard steel panel.

1.1.6 Procedure F measures shear adhesion of a filament reinforced tape when applied to a horizontal panel covered with NIST SRM 1810A standard fiberboard.

1.1.7 Procedure G measures the shear adhesion of a filament reinforced tape when applied to a horizontal panel covered with a standard fiberboard defined by CEN.

1.1.8 Procedure H measures the shear adhesion the same as Procedure A except the test is conducted at an elevated temperature and after a 10-min dwell time at the elevated temperature.

1.2 These procedures provide a means of assessing the uniformity of the adhesive of a given type of pressure-sensitive tape, usually tapes used for packaging applications. The assessment may be within a roll of tape, between rolls or production lots.

1.2.1 Variations in the tape backing and adhesive affect the response; therefore, these procedures cannot be used to pinpoint the specific cause(s) of nonuniformity.

<sup>1</sup>This test method is under the jurisdiction of ASTM Committee D10 on Packaging and is the direct responsibility of Subcommittee D10.14 on Tapes and Labels.

Current edition approved April 1, 2006. Published April 2006. Originally approved in 1978. Last previous edition approved in 2002 as D 3654/D 3654M - 02.

1.2.2 This test method is intended to replace AFERA 4012, CEN 1943, and PSTC (see 7.2).

1.3 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system are not exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems will result in non-conformance with the standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

### 2. Referenced Documents

#### 2.1 ASTM Standards:

A 666 Specification for Annealed or Cold-Worked Austenitic Stainless Steel Sheet, Strip, Plate, and Flat Bar  
D 3715/D 3715M Practice for Quality Assurance of Pressure-Sensitive Tapes

D 4332 Practice for Conditioning Containers, Packages, or Packaging Components for Testing  
D 5750/D 5750M Guide for Width and Length of Pressure-Sensitive Tape

E 122 Practice for Calculating Sample Size to Estimate, With a Specified Tolerable Error, the Average for a Characteristic of a Lot or Process  
2.2 AFERA Standard:  
AFERA 4012 Self Adhesive Tapes - Measurement of Static Shear Adhesion<sup>3</sup>

#### 2.3 CEN Standard:

EN 1943 Self-Adhesive Tapes—Measurement of Static Shear Adhesion<sup>4</sup>

<sup>2</sup>For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For Annual Book of ASTM Standards volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup>AFERA (Association des Fabricants Européens de Rubans Auto-Adhésifs), LAM, Iaan Copes van Cattenburch 79, NL-2858 EW, the Hague, the Netherlands.

<sup>4</sup>EN (European Norm), available from COMITE Européen de Normalisation, CEN Rue de Stassart, 36, B-1030, Brussels, Belgium.

## EXHIBIT III

## D 3654/D 3654M - 06

2.4 *PSTC Standard:*

PSTC-7 Holding Power of Pressure-Sensitive Tapes<sup>5</sup>

## 3. Summary of Test Method

3.1 *Procedure A, Shear Adhesion to Standard Steel Panel*—A strip of tape is applied to a standard steel panel under controlled roll down. The panel is mounted vertically, a standard mass is attached to the free end of the tape and the time to failure is determined.

3.2 *Procedure B, Shear Adhesion to a Standard (NIST) Fiberboard*—A strip of tape is applied to a panel covered with NIST SRM 1810A fiberboard under controlled roll down. The panel is mounted vertically, a standard mass is attached to the free end of the tape and the time to failure determined.

3.3 *Procedure C, Shear Adhesion to a Standard CEN Fiberboard*—A strip of tape is applied to a panel covered with the CEN standard fiberboard under controlled roll down. The panel is mounted vertically, a standard mass is attached to the free end of the tape and the time to failure is determined.

3.4 *Procedure D, Shear Adhesion to a Fiberboard with Controlled Roll Down*—The panel is mounted vertically, a standard mass is attached to the free end of the tape and the time to failure is determined.

3.5 *Procedure E, Shear Adhesion to a Standard Steel Panel*—A strip of filament reinforced tape is applied to a standard steel panel with a 120° bend at one end with controlled roll down. The panel is mounted horizontally, tape side up, with the free end of the tape allowed to hang vertically over the rounded end. A standard mass is attached to the free end of the tape and allowed to act for the specified time.

3.6 *Procedure F, Shear Adhesion of Filament Reinforced Tape to a Standard (NIST) Fiberboard*—A strip of filament reinforced tape is applied to a panel with a 120° bend, covered with NIST SRM 1810A standard fiberboard under controlled roll down. The panel is mounted horizontally, tape side up, with the free end of the tape allowed to hang vertically over the round end of the panel. A standard mass is attached to the free end of the tape and allowed to act for a specified time.

3.7 *Procedure G, Shear Adhesion of a Filament Reinforced Tape to a CEN Standard Fiberboard*—A strip of filament reinforced tape is applied to a panel covered with CEN standard fiberboard under controlled roll down. The panel is mounted horizontally, tape side up, with the free end of the tape allowed to hang vertically over the round end of the panel. A standard mass is attached to the free end of the tape and allowed to act for the specified time.

3.8 *Procedure H*—This procedure is conducted as described in Procedure A except the test is conducted at an elevated temperature after a 10-min dwell time at the elevated temperature.

3.9 For Procedures A, B, C, D, and H the preferred specimen size is 12 by 12 mm [0.5 by 0.5 in.]. A specimen size of 24 by 24 mm [1 by 1 in.] may be specified.

<sup>5</sup> PSTC (Pressure Sensitive Tape Council), 400 N. Michigan Ave., No. 2200, Chicago, IL 60611-4267.

3.10 For Procedures E, F and G the specimen width shall be 12 mm [0.5 in.]. For testing reinforced filament by Procedure H, the width shall be 12 mm [0.5 in.].

## 4. Significance and Use

4.1 Procedure A measures the ability of a pressure-sensitive tape to adhere to a standard steel panel under constant stress. This may or may not relate to the ability of the tape to adhere to other surfaces.

4.2 Procedures B, C, and D may be used to determine the shear adhesion of the tapes generally used to close fiberboard boxes in packaging applications.

4.3 Procedure D measures the shear adhesion of a pressure-sensitive tape to a nonstandard fiberboard, liner board, corrugated board, or other surfaces which is agreed upon for testing. This may be used to compare the shear adhesion of a tape to a particular fiberboard surface or to compare the shear adhesion of a tape to a variety of fiberboard surfaces.

4.3.1 The surfaces of similar fiberboards may exhibit considerable variation between mills, between batches from one mill, and within batches. Take care in the choice of samples and when comparing results between fiberboard surfaces which may not be exactly the same.

4.3.2 The precision of tests conducted on nonstandard surfaces may be different than that described in Section 13.

4.4 Procedures E, F, and G may be used to determine the ability of a filament reinforced tape to hold when placed under constant stress.

4.5 Procedure H may be used to compare the shear adhesion of tape applied to a standard steel surface and tested at an elevated temperature. The use of an elevated temperature during test tends to reduce the duration of the test.

## 5. Apparatus

5.1 *Specimen Cutter*<sup>5,6,7</sup>—The specimen cutter shall hold two single-edge razor blades in parallel planes, a precise distance apart, to form a cutter of exact specimen width. Two cutters, 12 mm [0.5 in.] and 24 mm [1 in.] cutting width, shall be available or appropriate alternates, which will not cause edge damage.

Note 1—The 12-mm [0.5-in.] cutter shall consist of a 12-mm [0.5-in.] thick by 200-mm [8-in.] length of aluminum bar stock 12-mm [0.5-in.] wide. The edges for about 125 mm [5 in.] from one end shall be rounded slightly to form a handle. The width of the bar for 75 mm [3 in.] shall be narrowed to exactly 12 mm [0.5 in.] minus the thickness of a single-edge razor (one of two used as cutting edges). The razor shall be held in position using side plates. The end of the cutter shall be cut away at a 45° angle to expose the cutting edges at one end of the blades. The edges shall be separated by 12 ± 0.10 mm [0.3 ± 0.005 in.]. The 24-mm [1 in.] cutter

<sup>6</sup> These widths correspond to the primary metric (SI) units described in Guide D 37.9/D 5750M. These so-called "modular metric" units generally are used throughout the world. If it is desirable to test slightly different widths (for example, 25 or 50 mm) of specimens per 8.4, this should be noted per 12.1.6 and calculations per 11.1 must account for the difference.

<sup>7</sup> The sole source of supply of the apparatus known to the committee at this time is Consultants International, 9349 Hamilton Drive, Mentor, OH 44061-1118. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

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shall follow the same description except the bar stock shall be 24-mm [1-in.] wide and shall be narrowed to exactly 24 mm [1 in.] minus the thickness of a single edge razor.

**5.2 Dispensing System**, for solvents, such as a wash bottle.  
**5.3 Panel**<sup>5,7</sup>:

5.3.1 For Procedures A, B, C, D, and H, a 50 by 125 mm [2 by 5 in.] not less than 1.1 mm [0.043 in.] thickness 302 or 304 stainless steel sheet with bright annealed finish in accordance with Specification A 666. The surface roughness height shall be  $50 \pm 25 \text{ nm}$  [ $2.0 \pm 1.0 \mu\text{in.}$ ] arithmetical average deviation from a mean line. One or both of the panel ends shall be ground to form a 90° angle with the panel surface. Panels showing stains, discolorations, or numerous scratches are not acceptable. New panels should be cleaned before use as described in 10.1, except with ten washes of the final solvent. Between uses, the panels test surface shall be protected from scratches and contamination, and the panels stored at conditions described in 8.1.

5.3.2 For Procedures B, F, and G, a panel as described in 5.3.1 shall have a 12 mm [0.5 in.] length at one end of the panel bent through an arc of 120° away from the test surface. The radius of the curvature of the finished surface at the bend shall be 1.5 to 3 mm [ $\frac{1}{16}$  to  $\frac{1}{8}$  in.].

**5.4 Roller**, mechanically or hand-operated.<sup>5,7</sup>

5.4.1 A steel roller  $85 \pm 2.5 \text{ mm}$  [ $3.25 \pm 0.1 \text{ in.}$ ] in diameter and  $45 \pm 1.5 \text{ mm}$  [1.75 by 0.5 in.] in width, covered with rubber approximately 6 mm [0.25 in.] in thickness, having a Shore scale A durometer hardness of  $80 \pm 5$ . The surface shall be a true cylinder void of any convex or concave deviations. The mass of the roller shall be  $2040 \pm 45 \text{ g}$  [ $4.5 \pm 0.1 \text{ lb}$ ].

5.4.2 No part of the apparatus shall increase the mass of the roller during use. The roller shall move either mechanically or by hand at the rate of  $10 \pm 0.4 \text{ mm/s}$  [ $24 \pm 0.5 \text{ in./min}$ ].

**5.5 Test Stands and Ancillary Apparatus:**<sup>5,7</sup>

5.5.1 **Procedures A, B, C, D, and H**—A test stand that shall hold the test panel, with tape applied, at an angle of 0 to 2° with the vertical, so that when the mass is acting on the test specimen, no peel forces will be exerted on the tape.

5.5.2 **Procedures E, G, and F**—A test stand that will support the test panel in a horizontal plane, approximately 300 mm [12 in.] above the work surface.

5.5.3 **Clamp or Hook**, that will allow attachment of the mass to the specimen, distributing the load equally across the tape specimen width.

**5.5.4 Test Masses:**

5.5.4.1 **Procedures A, B, C, D, and H**—The test mass shall be  $1000 \pm 5 \text{ g}$  or other mass as specified. The mass of the clamp or hook described in 5.5.3 shall be included as part of the total mass.

5.5.4.2 **Procedures E, F, and G**—The test mass shall be  $4.5 \pm 0.2 \text{ kg}$  [ $10 \pm 0.5 \text{ lb}$ ] or other mass as specified. The mass of the clamp or hook as described in 5.5.3 shall be included in the total mass.

**5.5.5 Timing Systems:**

5.5.5.1 **Procedure A, B, C, D, and H** to measure the interval in minutes, between the application of the load to the specimen and its separation from the panel.

5.5.5.2 **Procedures E, F, and G**, a suitable means of measuring the amount of slippage of the tape to 1 mm [ $\frac{1}{32}$  in.] on the panel after the mass has acted for 48 h.

**6. Reagent Materials**

**6.1 Purity of Reagents**—Reagent grade chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

**6.2 Solvents:**

6.2.1 Any of the following solvents may be used for cleaning.

**NOTE 2:** Before selecting or using these solvents for cleaning test panels be sure to read and follow all precautions on the chemical Material Safety Data Sheets (MSDS) and consult with Environmental, Health and Safety (EHS) Professionals.

6.2.1.1 **Dimethyl alcohol** nonresidual, technical grade or better.

6.2.1.2 **Methanol** (95%).

6.2.1.3 **Methyl Ethyl Ketone** (MEK).

6.2.1.4 **n-Heptane**.

6.2.1.5 **Acetone**.

6.2.2 For referee testing, the final cleaning shall be with MEK or acetone.

**6.3 Cleaning Material**, absorbent, surgical gauze, cotton wool, or tissue. To be suitable, materials must be lint free during use, absorbent, contain no additives that are soluble in the solvents listed in 6.2, and made exclusively from virgin materials.

**7. Sampling**

**7.1 Acceptance Sampling**—Sampling shall be in accordance with Practice D 3715/D 3715M. For Procedures A, E, and H three replicate specimens shall be averaged in accordance with Section 11 for each test result. For Procedures B, C, D, and G five replicate specimens shall be averaged. No single value shall be considered as representative of the roll under test.

**7.2 Sampling for Other Purposes**—The sampling and the number of test specimens depends on the purpose of the testing. Practice E 122 is recommended. It is common to test at least five specimens of a particular tape. Test specimens should be taken from several rolls of a tape and, whenever possible, among several production runs of a tape. Strong conclusions about a specific property of a tape cannot be based on test results of a single unit (roll) of product.

**8. Test Specimen**

**8.1 Removal from Roll:**

8.1.1 Unwind and discard at least three but no more than six outer wraps of tape from the roll before taking specimens for testing.

8.1.2 For Procedures A, B, and H, remove three specimens per sample roll for each test to be performed. For procedures B, C, D, and G remove five specimens per roll for each test to be performed. Remove specimens from freely rotating roll at the rate of 500 to 750 mm/s [20 to 30 in./min]. Where width or other factors causing high adherence to backing make it impossible to remove the specimen at the prescribed rate, remove it at a rate as close to 500 mm [30 in./min] as possible.

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8.2 When the tape is wider than the specimen specified in the test procedure, cut the specimen using the specimen cutter described in 5.1 from the center of the strip removed from the roll in accordance with 10.1.

8.3 Apply specimen within five min after unwinding.

8.4 *Test Specimen Size:*

8.4.1 Procedures A, B, C, D, and H, the test specimens shall be  $12 \pm 0.05$  mm [ $0.5 \pm 0.003$  in.], or other width, as specified (24 mm [1 in.] may be used) and approximately 150 mm [3 in.] long.

8.4.2 Procedure B, F, and G specimens shall be  $12 \pm 0.05$  mm [ $0.5 \pm 0.016$  in.] in width and approximately 300 mm [12 in.] long.

9. **Conditioning**

9.1 Condition rolls of tape in the standard conditioning atmosphere of  $23^\circ\text{C}$  and 50 % relative humidity as described in Practice D 4332 for no less than 24 h. Unless otherwise specified, test at these conditions.

9.2 Handle the panel as little as possible to minimize heat transfer from hands to the panel.

10. **Procedure**

10.1 For Procedures B, C, D, F and G, apply, by means of a double-coated pressure-sensitive tape, a piece of fiberboard (see 3.3.2, 3.3.3, 3.3.4, 3.3.6 and 3.3.7) wider and longer than the width and length of the test specimen, centered at one end of the test panels (see 5.3.1 and 5.3.2).

*Note 3*—Take care that the fiberboard is applied with the proper side up and with the grain of the paper perpendicular to the long direction of the test panel.

10.2 *Procedure A:*

10.2.1 Dispense one of the solvents listed in 6.2.1 onto the panel, wiping to dryness with fresh absorbent cleaning material (see 6.3). Repeat for a total of three washes with this solvent. Do not touch cleaned panel surfaces with fingers. *Warning*—All operations with solvents should be conducted in a well-ventilated hood.

*Note 4*—Discard panels showing stains, discoloration, or many scratches. During storage, panels should be protected from damage or contamination.

10.2.2 Center the test specimen on the 50 mm [2 in.] dimension at one end of the test panel and apply, without added pressure, to cover an area exactly 12 by 12 mm [0.5 by 0.5 in.], with tape. It may be desirable to mask the exposed adhesive of the free end of the specimen.

10.2.3 To prevent cutting of the specimen by the end of the panel during roll down, place another panel of the same or slightly lesser thickness and as wide as the test panel, under the free masked end of the specimen, and in contact with the end of the panel prior to roll down. Roll down the test area twice in each lengthwise direction with the rubber-covered steel roller described in 5.4.

10.2.4 Individually prepare each specimen and test within one min.

10.2.5 Place clamp or hook on the free end of the tape specimen, ensuring that the clamp or hook extends completely across the width of the specimen and is aligned to uniformly distribute the load.

10.2.6 Place the test assembly in the test stand so that the free end of the test specimen is vertical, ensuring that no peel forces act on the specimen.

10.2.7 Apply the 1000 g mass to the clamp or hook gently so as to cause no shear impact force on the tape specimen. Record the time elapse in which the tape specimen has separated completely from the test panel (see Fig. 1).

10.3 *Procedures B, C, and D*—Conduct these tests as described in 10.2, except the test panel shall be covered with fiberboard as described in 10.1.

10.4 *Procedure G:*

10.4.1 Clean, as described in 10.2.1, a test panel described in 5.3.1.

10.4.2 Apply one end of the specimen, about 100 mm [4 in.] in length, adhesive side down, to the longitudinal surface of the test panel. The tape must be at a true right angle to the bent

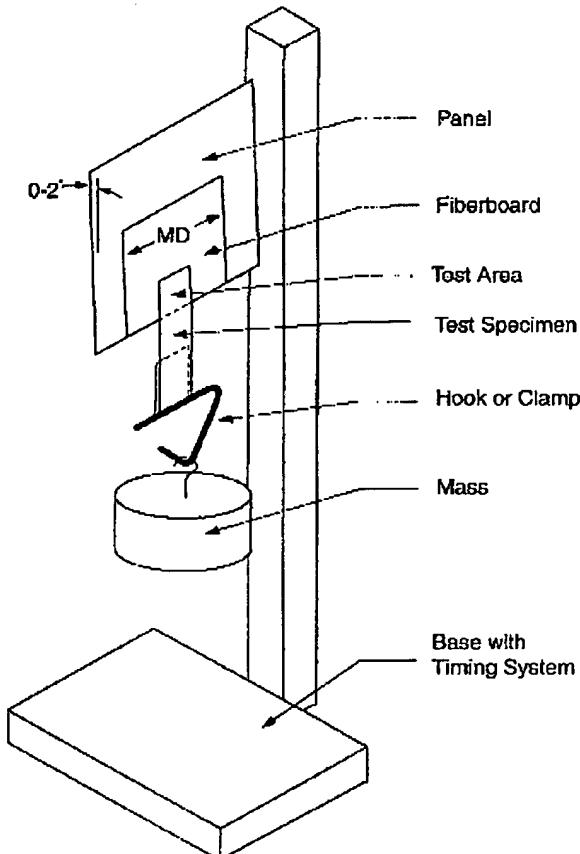


FIG. 1 Sketch of Typical Shear Adhesions Tester for Procedures B, C, and D. (For Procedures A and H, substitute steel panel instead of fiberboard.)

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edge of the panel. Allow the remaining 200 mm [8 in.] to extend over and beyond the bend edge of the panel.

10.4.3 Using a square, cut across, and through the width of the tape specimen 75 mm [3 in.] back from the front of bend in the horizontal plane of the test panel surface.

10.4.4 Roll twice, once in each lengthwise direction using the rubber covered steel roller described in 5.4.

10.4.5 Place the clamp or hook on the free end of the specimen, ensuring that it extends completely across the width of the specimen, and is aligned to distribute the load uniformly.

10.4.6 Place the test assembly in the test stand so that the panel is horizontal, tape side up, and the free end of the test specimen is vertical. Apply the 1.5-kg [10-lb] mass to the clamp or hook gently so as to cause no impact force on the specimen (see Fig. 2).

10.4.7 At the end of 48 h under load, examine the specimen for evidence of slippage. Measure any slippage that has occurred to the nearest 1 mm [ $\frac{1}{32}$  in.].

10.5 *Procedure F and G*—Conduct these tests as described in 10.4, except the test panels shall be covered with fiberboard as described in 10.1.

10.6 *Procedure H*:

10.6.1 Prepare test specimens as described in 10.2.1-10.2.5.

10.6.2 Place the test stand, with specimen in place, in an oven maintained at 50°C [120°F].

10.6.3 Allow to condition at 50°C [120°F], for ten min, then apply 1000-g mass to the clamp or hook gently so as to cause no shear impact force on the tape specimen. Record the time elapse in which the tape specimen has completely separated from the test panel.

## 11. Calculation

### 11.1 Acceptance Sampling:

11.1.1 *Procedures A, B, C, D, and H*—To determine the test results for each roll of tape, convert each of the test results (time to failure) to its common or natural logarithm. Obtain the arithmetic mean of all logarithms and then convert back to time

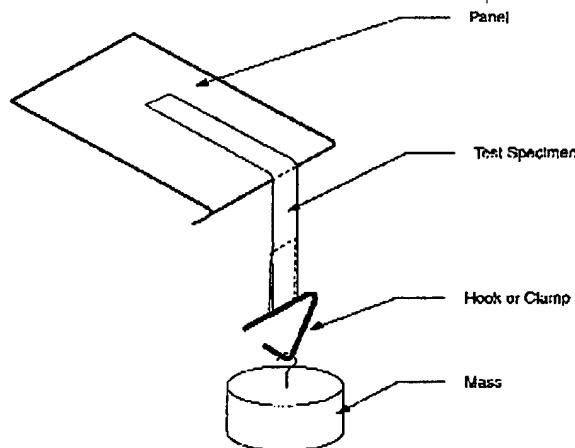


FIG. 2 Sketch of Typical Shear Adhesion Tester for Procedure E.  
(For Procedures F and G, substitute a fiberboard surface.)

by obtaining the appropriate antilogarithm. This gives the test results for the roll of tape under consideration in the sampling plan.

11.1.2 *Procedures E, F and G*—It is common to interpret the results of these test methods as passing or failing a preset acceptance criterion.

11.2 *Other Purposes*—Convert the test results (time to failure) from each specimen to its common or natural logarithm. Conduct an analysis of the logarithm of the data to determine the desired descriptive or comparative statistics. The resulting means and confidence intervals may be converted back to the original time units by obtaining the appropriate antilogarithm. Use of the Weibull distribution is an acceptable alternative. Some computer programs for data analysis allow the use of right-censored data. These censored data may have a time to termination rather than a time to failure.

## 12. Report

### 12.1 Report the following information:

12.1.1 Statement including which test procedure was used and indicating any deviation from the procedure as written,

#### 12.1.2 Identify source of each roll,

12.1.3 For Procedures A, B, C, D, and H the time for the tape to separate completely from the panel. For Procedures E, F, and G the amount of slippage in mm (in.) to the nearest mm [ $\frac{1}{32}$  in.],

#### 12.1.4 Dwell time, if other than one min,

12.1.5 Test specimen size for Procedures A, B, C, D, and H if other than 12 by 12 mm [0.5 by 0.5 in.]. Width of specimen for Procedures E, F, and G if other than 12 mm [0.5 in.],

12.1.6 Conditioning if other than 23°C [73.4°F] or 50 % RH,

12.1.7 Test temperatures for Procedures A, B, C, D, and G if other than 23°C [73.4°F] and test temperature for Procedure H, if other than 50°C [120°F],

12.1.8 *Mode of Failure - Cohesion (Cohesive strength, internal bond)*—The ability of the adhesive to resist splitting. Good cohesion is necessary for clean removal. Adhesion is a bond produced between a pressure-sensitive tape adhesive and a surface for Procedures A, B, C, D, and H; and,

#### 12.1.9 Fiberboard substrate, if Procedure D is used.

## 13. Precision and Bias

13.1 *Procedures A, B, C, and D*—Repeatability (within laboratory variability) information is available from one laboratory; several types of tape were evaluated using time to failure in minutes converted to natural logarithm for analysis. Examples of averages and within-roll standard deviations (including test error) are as follows:

Tape	X	Converted X, min.	Within Roll
A	8.74	8248	0.39
B	8.27	3905	0.23
C	2.43	111	0.27
D	8.50	665	0.45
E	5.99	395	0.82

13.1.1 The remaining components of variance are expected to be similar to those for Procedure H. The between roll

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standard deviation and the residual (including within roll) standard deviations will vary, depending on the tape type and the manufacturer.

13.1.2 *Procedures E, F, and G*—No statement is made about the precision of the procedures since results merely state whether there is conformance to specified criteria for success.

13.1.3 *Procedure H*—An interlaboratory evaluation of two types of pressure-sensitive tapes by three laboratories has been conducted for Procedure H. The results are available.<sup>8</sup> It is based on an evaluation using lognormal distribution; the times to failure in minutes were converted to their natural (base e) logarithms for analysis. The tapes tested in this study had a grand mean of 1.2, which converts to 3.3 min. The components of the variance for the following factors were estimated: between operator in a laboratory, between rolls of tape, and residual (including test error and within roll variation).

\* Supporting data are available from ASTM Headquarters. Request Rpt. D-10-1002, Report 2.

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13.1.3.1 A summary of the pooled standard deviation is as follows:

Between laboratories	0.46
Between testers	0.247
Between rolls	0.185
Residual	0.411

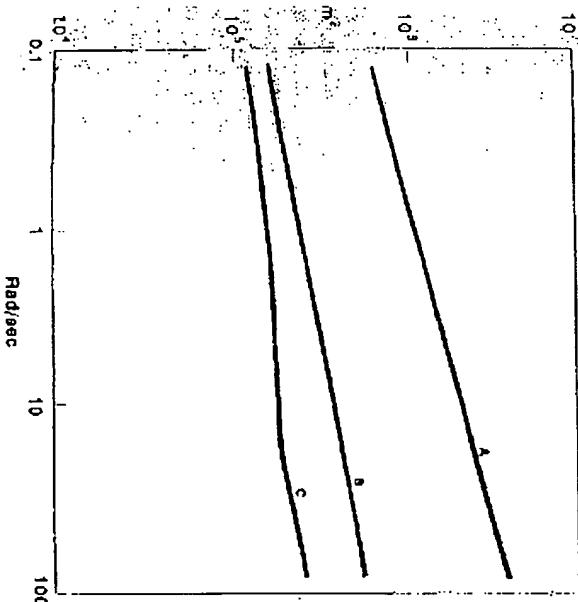
13.1.3.2 These components may be combined in several ways to obtain the desired repeatability (within laboratory) and reproducibility (between laboratory) estimates of precision. One company reports that, with care, the between laboratory standard deviation and the residual (including within roll) standard deviation may be higher or lower than reported here, depending on the tape type and manufacturer.

13.2 *Bias*—No measurement of bias is possible with this test method because an accepted reference or referee value is not available.

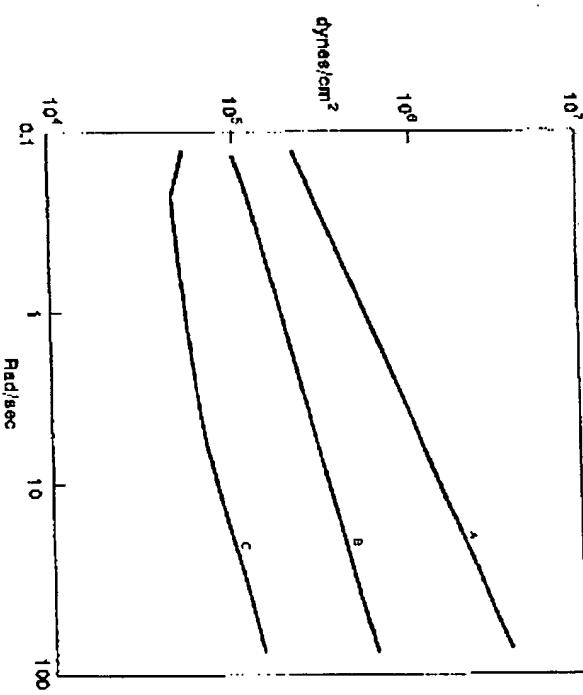
## 14. Keywords

14.1 filament-reinforced tape; pressure-sensitive tape; shear adhesion

of the mechanical properties of a truly removable adhesive is more easily assessed without experimental investigation. Figure 10-23 shows the results of removable adhesives [21]. Curve A represents an elastic low peel adhesion product. Low tack is indicated by high  $G'$  at low frequencies. Curve B represents  $G'$  of a standard acrylic adhesive shown for purposes. Its  $G'$  values are much lower at low frequencies and exhibits a relatively high tack, such as expected from a regular pressure sensitive adhesive. Curve C represents  $G'$  curve of a soft, high tack adhesive of low peel adhesion and removable. This adhesive has a low  $G'$  at high frequencies. The slope of the  $G'$  curve is also lower than that of curves A and B. A low  $G'$  value at high frequencies suggests low peel adhesion and that it is easier to remove a pressure sensitive adhesive label. Loss values shown in Figure 10-24 lead to the same conclusions as the  $G'$  curves.

Fig. 10-23. Storage modulus ( $G'$ ) of removable adhesives.

- A. Elastic low tack adhesive.
- B. Standard acrylic adhesive.
- C. Soft high tack adhesive.

Fig. 10-24. Loss moduli ( $G''$ ) of removable adhesives.

- A. Elastic low tack adhesive.
- B. Standard acrylic adhesive.
- C. Soft high tack adhesive.

Limiting shapes of ideal  $G'$  curves for removable and high peel adhesives are shown in Figure 10-25. The level of  $G'$  at low frequencies is fixed due to the requirement that the adhesive must be tacky. Dahlquist's criterion suggests that the compressive modulus of a pressure sensitive adhesive cannot exceed  $10^7$  dynes/cm<sup>2</sup> in the time frame of the bonding process (at low frequencies) [22]. The removable of the adhesive is favored by a low peel adhesion i.e. a low  $G'$  at high frequencies. Thus a curve of zero slope (A) is the ideal for an easily removable adhesive. Such an adhesive may be impossible to compound, but this at least shows the direction to advance in the development of such adhesives.

In developing high peel adhesives we must attempt to increase  $G'$  at high frequencies as much as possible. Figure 10-25 curve B shows an idealized  $G'$  curve of such adhesive [21]. The value of  $G'$  at low frequencies is fixed by the requirement that the adhesive is tacky at the time of application. To realize the maximum peel adhesion we want to obtain as high a storage modulus at high frequencies as possible. While pressure sensitive adhesives do not exhibit very high moduli, they can be cured to harder non-pressure sensitive substances which can have a very high moduli. Such curing could be done after the adhesive tape is

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